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फेरोक्रोमियम का रासायनिक विश्लेषण

भाग 7 फेरोक्रोमियम/चार्जक्रोम में क्षारमितीय पद्धति द्वारा फासफोरस ज्ञात करना

Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 7 DETERMINATION OF PHOSPHORUS IN FERROCHROMIUM/ CHARGECHROME BY ALKALIMETRIC METHOD

ICS 77.100

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2003

Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 7) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Method for chemical analysis of ferro alloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungesten, while reviewing this standard the sectional committee had decided that a separate series for each ferroalloy be prepared. Accordingly IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemical analysis of ferromolybedenum is covered in a series of parts of IS 12614 'Method of chemical analysis of ferromolybdenum'. The chemical analysis of ferrochromium is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferrochromium in IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' will be superseded. This Standard (Part 7) covers the method for determination of phosphorus in ferrochromium/chargechrome by alkalimetric method. The other parts in this series are:

(Part 1) : 1992 Determination of silicon in high carbon ferrochromium by gravimetric method
(Part 2) : 1992 Determination of silicon in low carbon ferrochromium by gravimetric method
(Part 3) : 1992 Determination of phosphorus in low carbon ferrochromium by gravimetric method
(Part 4) : 1992 Determination of total sulphur in low carbon and high carbon ferrochromium by direct combustion method
(Part 5) : 1992 Determination of chromium in high carbon ferrochromium/chargechrome by Dichromate Titration
(Part 6) : 1997 Determination of chromium in low carbon ferrochromium

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 7 DETERMINATION OF PHOSPHORUS IN FERROCHROMIUM/ CHARGECHROME BY ALKALIMETRIC METHOD

1 SCOPE

This standard (Part 7) describes the method for determination of phosphorus in the range of 0.01 to 0.06 percent.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision*)
1472 : 1977	Methods of sampling of ferroalloys for determination of chemical composition (*first revision*)

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1472.

4 QUALITY OF REAGENTS

Unless specified otherwise analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF PHOSPHORUS

5.1 Outline of the Method

Sample is digested with HCl and HNO_3 acid and fumed with perchloric acid. During fuming P & Cr are oxidized. Subsequently oxidized chromium is removed as chromylchloride on addition of hydrochloric acid. Phosphorus present as orthophosphate is precipitated as ammonium phosphomolybdate and determined by alkalimetric titration.

5.2 Reagents

5.2.1 *Concentrated Nitric Acid,* rd = 1.42 (conforming to IS 264).

5.2.2 *Hydrofluoric Acid 48 percent,* rd = 1.16.

5.2.3 *Perchloric Acid,* 60 percent.

5.2.4 *Concentrated Hydrochloric Acid,* Sp. Gr. 1.16 (conforming to IS 265).

5.2.5 *Ammonium Nitrate Solution*, 50 percent (*w/v*).

5.2.6 *Ammoniummolybdate Solution*

a) Weigh 50 g of ammoniummolybdate in a 500 ml beaker, add 100 ml of ammonium hydroxide, dissolve and make up to 200 ml. Keep it separately.

b) Add 400 ml of nitric acid to 400 ml of distilled water in a 1-litre conical flask, cool and keep it separately.

Slowly add solution a) into solution, b) with continuous stirring and cooling. Allow to settle and filter before use.

5.2.7 *Potassium Nitrate Wash Solution*, 2 percent (*w/v*).

5.2.8 *Standard Nitric Acid (0.075 N)* — Take 4.7 ml of concentrated nitric acid (rd = 1.42) to a 1-litre. volumetric flask and make to the mark. Standardize the solution against standard sodium carbonate solution using methylorange as indicator.

5.2.9 *Standard Sodium Carbonate Solution (0.075 N)* — Weigh 0.039 75 g preheat sodium carbonate from 250 to 270°C for half-an-hour. Cool in a desiccator, and transfer into a 100 ml volumetric flask. Dissolve in distilled water and make up to the mark.

5.2.10 *Methylorange* — Dissolve 0.05 g in 100 ml distilled water.

5.2.11 *Phenolphthalein* — Dissolve 0.5 g in 50 ml alcohol and dilute with 50 ml distilled water with constant stirring.

5.2.12 *Hydrochloric Acid* — 1 : 3 *v/v*

5.2.13 *Sodium Hydroxide Solution (Approximately 0.075 N)* — Dissove 3 g of sodium hydroxide in 1 litre of distilled water.

5.3 Procedure

5.3.1 Weigh 2 g dried and agated sample (for P 0.01 to 0.03 percent) or 1 g (for P 0.03 percent above) agated sample and transfer to 500 ml conical flask. Add 10 ml hydrochloric acid followed by 5 ml nitric acid and few drops of hydrochloric acid. Heat the solution a hot plate. After about 30 min, add 10 ml of perchloric acid and reflux for 30 min, after fuming starts. Again add 10 ml of perchloric acid and continue fuming on a hot plate till the mass almost dissolves. Cool the beaker and dissolve the salts with minimum quantity of water and transfer the contents into a conical flask of 500 ml capacity. Clean the bottom of the flask with a little concentrated hydrochloric acid and boil. Evaporate the solution on a hot plate till dense white fumes come out.

NOTE — Below -200 Mess preferably in case of high carbon ferrochrome.

5.3.2 Add concentrated hydrochloric acid (*see* **5.2.4**) drop wise till brown fumes come out. Keep on hot plate and again reflux for some time to reoxidize chromium. Again add hydrochloric acid to remove chromium.

5.3.3 Continue the operation (*see* **5.3.2**) till all chromium is expelled, that is, no more brown fumes on addition of hydrochloric acid.

5.3.4 Evaporate the bulk to syrupy condition. Dissolve the salts with about 50 ml of water. Add 25 ml of ammonium nitrate solution and 50 ml ammonium molybdate solution. Warm the content to 65°C with constant shaking. Remove from the heat and shake further for about 5 min. Phosphorus is precipitated as fine yellow crystal. Allow the precipitate to settle for 2 h atleast.

5.3.5 Filter the precipitate on a paper pulp pad, wash the flask and precipitate several times with cold neutral potassium nitrate wash solution until the washings tested no longer acidic to phenolphthalein (*see* Note). Transfer the precipitate with pad to the original flask where phosphorus was precipitated. Add 10 to 15 ml of distilled water and shake the flask to discipate the pad. Add 10 ml of sodium hydroxide solution to the flask and shake well. Titrate the excess sodium hydroxide solution with standard acid using phenolphthalein as indicator.

NOTE — 10 ml of final washing collected in a test tube should give pink colour with phenolphthalein by a drop of sodium hydroxide solution of approximately 0.075 N. Normally the precipitate is washable free from acid by giving about 5 to 6 washings.

5.3.6 Carry out a reagent blank with same quantity of all the reagents and chemicals used except the sample.

6 CALCULATION

$$\text{Phosphorus, percent} = \frac{(V_1 - V_2)\, N \times 0.001\,35 \times 100}{M}$$

where

V_1 = volume, in ml, of standard nitric acid required by the blank;

V_2 = volume in ml, of standard nitric acid required in the test sample;

N = normality factor of standard nitric acid used; and

M = mass, in g, of the sample taken.

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